

INFORMATION REPORT INFORMATION REPORT

CENTRAL INTELLIGENCE AGENCY

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1. In November 1963, the USSR was offering abroad licenses on the following chemical processes:
- a. Process for the production of weak nitric acid.
  - b. Continuous process for the production of alkoxysilanes.
  - c. Process for the production of hydrogen peroxide.

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### PROCESS FOR THE PRODUCTION OF WEAK NITRIC ACID

The proposed process allows the production of nitric acid with a monohydrate concentration of 47-55 %.

The process requires, as starting material for the production of weak nitric acid, synthetic ammonia and air, with both these materials being previously purified, in addition to which the air is subjected to water scrubbing.

The chemism of the process resides in the catalytic oxidation of ammonia by air under atmospheric pressure and in the absorption of nitric oxides under a pressure of 3.5 - 4 atm. Gaseous as well as liquid ammonia may be used, while atmospheric air is utilised for the oxidation of ammonia.

The proposed process for the production of weak nitric acid is characterised by the following outstanding features :

1. The process whereby nitrous gases are cooled prior to compression has been coupled with the process of scrubbing ammonia salts away from the gases. Both processes are being carried out in the same apparatus.

2. In contrast to other methods wherein heat resulting from the oxidation of nitrous gases is drawn away by way of cooling water, the proposed process calls for the oxidation of compressed gases within an uncooled and insulated oxidising device with the oxidation reaction heat being accumulated by nitrous gases, which raises considerably their temperature.

This heat serves to warm up the gases flowing into the recuperation turbine which brings about a high degree of recuperation energy that is being consumed in compressing the nitrous gases (the recuperation percentage being about 40-45%).

3. Instead of packed or bubbling absorbers, an absorption tower featuring sieve plates especially perforated for the purpose is being used for carrying out the absorption process. This tower is especially designed to insure a considerably widened phase contact

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surface which imparts a high speed to the mass transfer. Owing to the above, the entire process of acid synthesis is considerably speeded up and the degree of nitric oxide absorption thus achieved is considerably higher than when proceeding by way of hooded towers designed for bubbling system operation.

4. The process calls for an equipment consisting of control, measuring and self-regulating instruments and blocking devices designed to insure continuity, stability and safety of operation as well as accident-free stoppage of the installation with an operating staff reduced to a minimum.

The chart reproduced herebelow shows the technical and economical data arrived at by comparing the proposed process with well-known processes designed for the production of nitric acid under atmospheric pressure (1.1 atm) and under a pressure of 8-9 atm :

| Denomination                         | Unit of Measure                                  | M e t h o d |                |                |
|--------------------------------------|--|-------------|----------------|----------------|
|                                      |  | 3.5 at.     | 1.1 at.        | 8-9 at         |
| I. Capital Expen-<br>diture :        |  |             |                |                |
| a) in terms of stainless steel       | T/T of HNO <sub>3</sub><br>per 24 hrs.           | 1.05        | 1.95           | 1.13           |
| b) in terms of ce-<br>ramic rings    | T/T of HNO <sub>3</sub><br>per 24 hrs            | nil         | 18.6           | nil            |
| c) investments of capital            | %  | 100         | 110            | 140            |
| II. Exploitation. ex-<br>penditure : |  |             |                |                |
| a) ammonia consum-<br>ption          | T/T of HNO <sub>3</sub><br><del>per 24 hrs</del> | 0.283       | 0.285          | 0.292          |
| b) catalyst consum-<br>ption         | G/T of HNO <sub>3</sub>                          | 0.049       | 0.06-<br>0.049 | 0.25-<br>0.175 |
| c) electric power<br>consumption     | VTCH/T   | 200         | 125            | 380            |

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One ton  $\text{HNO}_3$  requires, under five process lines, 0.15 man-hour and 0.37 man-hour under two process lines. The output of one process line is 135 T  $\text{HNO}_3$  monohydrate per 24 hours. The total yield in terms of nitrogen is 95-96 %.

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CONTINUOUS PROCESS FOR THE PRODUCTION OF ALKOXYSIANES

The proposed method allows the production of high purity compounds used in the manufacture of semi-conductor materials.

Well-known methods, used presently in the production of alkoxyislanes, have been hitherto based on the interaction reaction of appropriate chlorosilanes with alcohols, causing hydrochloric acid gas to be released, while a side reaction takes place, involving hydrochloric acid and the end product - alkoxyislane - with chloroether being formed and resulting in a substantial lowering of the alkoxyislane yield. The maximum product yield, when applying well-known processes, has been amounting to 40-45 %.

The proposed process allows a yield figure ranging from 80 % to the theoretical. This results is arrived at through a determined selection of the interrelation of the compounds, optimum temperature and the high speed with which hydrochloric acid gas is removed from the reaction mixture. In this way, the conditions of synthesis and desorption that have been found, ensure an increase of the yield of the end product by 35-45 %.

The process for the synthetic of alkoxyislanes is being carried out in a reactor, allowing a rapid and complete blending of the compounds under the required temperature and a high intensity of the mass exchange during the desorption of the hydrochloric acid gas.

The output of the reactor is equal to 700 kg./hour per 1 cubic meter capacity with the apparatuses working without interruption. These apparatuses are compact in design, featuring small sizes and are easy to build, being made of glass, porcelain, nickel-molybdenum alloy, enamelled steel. Separate elements of the apparatuses may be made of fluoroplastics, vinylplastics and plexiglass.

The production of alkoxyislanes in accordance with the proposed process is characterized by the following material and utilities requirements calculated for 1 kg of completed product :

|                     |         |
|---------------------|---------|
| Chlorosilane.....   | 1.2 kg  |
| Alcohol.....        | 1.6 kg  |
| Steam.....          | 6.0 kg  |
| electric power..... | 0.6 kv. |

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PROCESS FOR THE PRODUCTION OF HYDROGEN PEROXIDE

Hydrogen peroxide is widely used as an active oxidizing agent in the textile and food industries, in the production of building material, in medicine, for organic synthesis purposes and in a number of other industrial applications.

The anthraquinone process for the production of hydrogen peroxide is known to be the most economical of all the methods devised for industrial purposes. It consists in the alkylanthraquinone - or a mixture of alkylanthraquinones - being subjected to a catalytic hydrogenation in an organic solvent medium, followed by an oxidation through oxygen.

The reduction and oxidation reactions are carried out within the organic solvent medium, intended to dissolve anthraquinone. The efficiency of the process, as a whole, depends on both the chemical activity and the solubleness of the anthraquinone, as well as on the activity and the selectivity of the hydrogenation catalyst.

In order to ensure the required selectivity for the catalyst, it is subjected to a special treatment, which varies according to the conditions under which the hydrogenation process has been carried out and the properties of the anthraquinone selected for the production of hydrogen peroxide.

By using such a catalyst, it has been possible to increase the output of the apparatuses 1.5 - 2 times, thus reducing by 30-40 % the cost of producing hydrogen peroxide.

This makes it possible for the process to be carried out under the maximum high concentration of anthraquinones, allowing higher hydrogenation phase temperatures. The especially processed catalyst ensures an optimum proportion of mixed anthraquinones in the working solution.

This latter advantage almost eliminates the necessity of periodical changing - or extensive regeneration - of the working solution.

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Process for the production of hydrogen peroxide (cont'd)

By applying determined proportions of anthraquinone and a properly processed catalyst, a solution is obtained with a hydrogen peroxide concentration of 26-45 % by weight.

This new process does not require any intricate equipment; all the apparatuses intended for the hydrogenation, oxidation, extraction and drying of the working solution are made of aluminium and steel.